ious laboratories around the world. Clearly the technical difficulties connected with the requirements on the quality of the electron bunches (emittance, peak current, etc.) grow dramatically as the required wavelength decreases towards the range of the hard x-rays.

In the pursuit of the higher brilliance of fourth generation sources there is an alternative road to the Linac FEL's . This is the approach based on recirculating Linacs, as exemplified by the 4GLS ("4th Generation Light Source") project being designed at Daresbury Laboratory in the UK. The idea is to combine the "few passes" strategy to lower emittance with the advantage of the ring geometry in which a large number of tangential beam lines and experimental stations can be exploited by users in parallel. It consists of a Linac that feeds electrons into one or several arc-shaped sections on which undulators, wigglers and even FEL's can be inserted (Fig. 4). At the end of the (last) arc, electrons are either dumped or fed back into the Linac, where they can return part of their energy to the RF fields in the accelerating cavities (this latter version is called "energy-recovery Linac"). The Linac is preferably of the superconducting type, one of the reasons being that it can support a much higher repetition rate (a much smaller time interval between successive bunches).

Conclusions

The interest of scientists from a variety of disciplines in ever more brilliant light sources in the UV and x-ray spectral region is not decreasing. While the third generation sources are still delivering a wealth of interesting science, the intense activity to generate spatially coherent, ultrashort pulses promises a new frontier to be opened a few years from now. It will be extremely interesting to monitor the progress of the many projects underway all over the world.

About the author

Massimo Altarelli was trained at the University of Rome as a condensed matter theorist. At the ESRF in Grenoble, he was Research Director (1987-1993), and Head of the Theory Group (1994-1998). Since 1999 he has been in Trieste on the staff of the International Centre for Theoretical Physics, and Director of the Elettra Synchrotron Light Source.

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[1] An excellent account of the early history of synchrotron radiation, with many references, can be found on pages 27-35 of E.-E. Koch, D.E. Eastman and Y. Farge, "Synchrotron radiation – a powerful tool in science", in *Handbook of Synchrotron Radiation*, vol 1A, E.-E. Koch editor (North Holland, Amsterdam, 1983) pp 1-63;

and also at the site http://xdb.lbl.gov/Section2/Sec_2-2.html, which provides an account by A.L. Robinson, as part of the X-ray Data Booklet Project of Lawrence Berkeley National Laboratory.

[2] See http://www-ssrl.slac.stanford.edu/jhhome.html for a short description of the state of the project

[3] J. Rossbach, Nucl. Instr. Meth A475, 13 (2001); for a first experimental application, see H. Wabnitz *et al.*, Nature 420, 482(2002)

Synchrotron radiation imaging and diffraction for industrial applications

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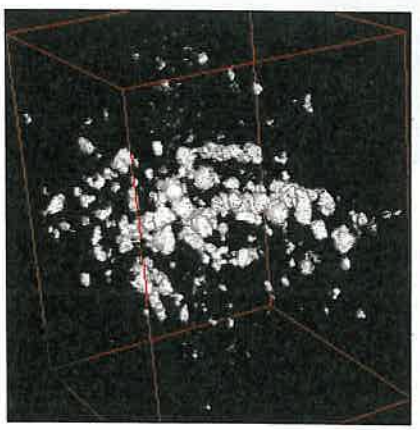
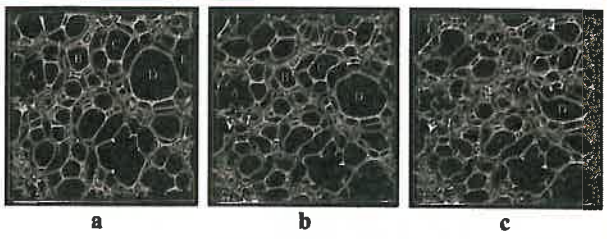
The use of the modern synchrotron radiation (SR) sources for X-ray imaging and diffraction provides new possibilities for applied science and technology. They include an enhanced resolution (spatial and/or temporal), the possible use of the coherence of the beam, in-situ experiments and accurate quantitative measurements of local density or strain. Selected applications showing the improved capabilities in absorption or phase microtomography, time-resolved diffraction experiments showing the way a lithium-based battery works, or strain measurements on stir-welding materials, are presented as examples. The combination of diffraction and imaging (i.e. diffraction with micron spatial resolution) allows further applications, such as the study of grain growth, or the X-ray topographic investigation of defects responsible for spurious modes in single crystal resonators.

The aim of the present paper is to highlight the contribution of synchrotron radiation X-ray imaging and diffraction to the investigation of industrial topics. These techniques, which allow the visualization of the volume of systems opaque for other probes and the determination of their structure, have been dramatically renewed by the use of modern, "third generation", synchrotron radiation (SR) facilities beams. Real time/high-resolution experiments are performed for both X-ray imaging and diffraction. The high energy of the available beams allows the deformation within bulk materials to be investigated. The high coherence of the beam allows phase contrast imaging to be exploited, the contrast arising from phase variations across the transmitted beam. Absorption and phase microtomography provide three-dimensional information on features like cracks, porosities or inclusions, which are of high interest for applied topics such as metallurgy, polymers, or reservoir rocks containing fuel. The spatial resolution of these techniques is now in the 1 µm range. Lastly the combination of diffraction and imaging, through the new tracking techniques, or the use of Bragg diffraction imaging (X-ray topography) provides additional information on industrially interesting materials.



◀ **Fig. 1:** 3D image of the distribution of holes (in white) within a copper target (transparent), 269*284*201 pixels, 2 µm voxel size



▲ Fig. 2: Reconstruction of an open-cell flexible polyurethane foam at several levels of compressive strain. (a) 0%, (b) 10%, (c) 23%, these percentages being the ratio of the variation of vertical length of the foam over the original, unstrained, length. The 3D renditions represent volumes of 7mm × 7mm × 1.4 mm.

The original features of third generation synchrotron radiation facilities such as the APS, ESRF, or Spring 8, for imaging and diffraction applications, are:

a) the very high intensity of the X-ray beam (factor 10^6 with respect to usual X-ray generators); both white and monochromatic beams can be used
b) the availability of photons spanning the whole range from the infrared to hard X-rays (up to 300-400 keV)
c) the design of beamlines optimised for a given set of techniques
d) the small size of the electron beam cross-section (< 100 μm), which leads to high brilliance, and to a sizeable lateral coherence of the X-ray beam.

Absorption and phase microtomography

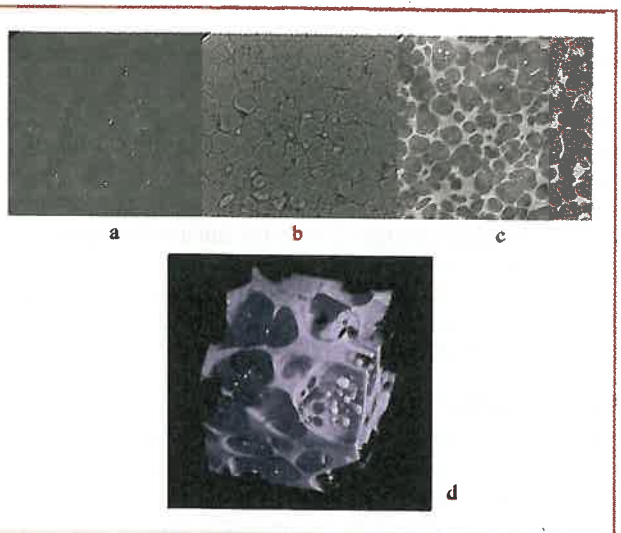
The principle of microtomography is very similar to that of the well-known medical scanner. When applied to materials investigation, it consists in recording a series of radiographs (typically of the order of 1000) for different angular positions of the sample, which rotates around an axis perpendicular to the beam. Several laboratory microtomographs have been commercially produced over the last years (Rüegsegger *et al.*, 1996, Sasov and Van Dyck, 1998). But the best images, in terms of spatial resolution, signal-to-noise ratio and quantitative exploitation, are obtained using synchrotron radiation. This results from the high intensity, practically parallel and monochromatic incoming beam. In this approach there is no image magnification, and the spatial resolution mainly results from the effective pixel size of the detector. The range of pixel sizes available at the ESRF goes from 0.3 μm to 30 μm, and a big effort is being produced to enhance the spatial resolution down to the 100 nm range. The total acquisition time is in the 10^{-2} ("fast tomography") to1 hour range, and the recorded data is often several Gigabytes.

An increasing number of applied or industrial research laboratories require the use of microtomography to solve some of the problems they encounter. To achieve this requirement they either buy beamtime and expertise, or have access to the synchrotron facilities through collaboration with Universities or research groups and peer-reviewed proposals. The industrial requirements include, most of the time, confidentiality, rapid access and full service. Most facilities, and in particular the ESRF, propose such a service, going from the experiment to the data analysis (volume reconstruction, extraction of relevant parameters ...).

Figure 1 is an example of such application-oriented investigation. A strong impact on a copper target creates porosity within the bulk metal. The investigation of the pore sizes and distribution is of high importance for applications. The only way to image these pores inside such a sample is X-ray microtomography (Bontaz-Carion *et al.*, 2001). The spatial distribution of pores within one of these copper samples was determined (figure 1), and shown to be less homogeneous, both from the point of view of size and of location, than what was expected from theoretical models.

Another example of absorption tomography, which implies a specially designed sample environment cell, is the in-situ investigation of an open-cell polyurethane foam at several levels of compressive strain (figure 2). This work, performed by a research group from ICI Polyurethane in collaboration with a group of the University of Cambridge, correlates the macroscopic behaviour (stress/strain curve) with the local structure modifications. It shows that the initial phase of compression, which is associated with a linear elastic response, corresponds to a bending of the struts, whereas the plateau in the stress/strain curve is linked to the collapse a whole band of cells (Elliott *et al.*, 2002). This result was known from surface observations, but no volume evidence was available.

The X-ray beams produced at third generation synchrotron radiation facilities exhibit a high degree of coherence. This results from the small source size σ (in the 50 μm range) and the large source to sample distance L (in the 100 m range). The transverse coherence length $d_c = \lambda L/2\,\sigma$, is in the 100 μm range, and allows "phase images" to be recorded by just varying the sample-to-detector distance ("propagation technique", reviewed for instance by Cloetens *et al.*, 1999a). The great advantage of this new type of imaging is the increased sensitivity it provides, either for light materials such as polymers, or for composites made up of materials with neighbouring densities (for example Al and SiC). A first use of the phase images relies on the visualisation of the phase jumps that occur at the edges of a particle or porosity imbedded in a matrix having a different index of refraction. Phase microtomography based on the visualisation of the edges was used, for instance, to understand the mechanisms of degradation in Al-SiC composites. It was possible not only easily to visualize the SiC reinforcing particles, but also to observe the nucleation and propagation of cracks when the material is submitted, *in situ*, to tensile stress. The cracks appear first in the elongated particles, and their number is 50% more than suggested by surface investigations (Buffière *et al.* 1999).



▲ Fig. 3: Tomographic images of an Al-Si alloy, quenched from the "semi-solid" state a) Detector to sample distance D = 0.7 cm b) D =60 cm c) "holotomographic" image, d) 3D rendering showing the former "liquid" phase (courtesy L. Salvo and P. Cloetens).

features

Phase imaging based on the detection of the edges does not allow the local phase to be extracted quantitatively, and its spatial resolution is limited by the occurrence of the fringes used to visualize the borders. A more quantitative approach of phase imaging and tomography was developed. It is based on the combination of several images recorded at different distances. An algorithm, initially developed for electron microscopy by the Antwerp group, was successfully adapted to the X-ray case, and allows the "holographic" reconstruction of the local phase, well beyond the images of edges (Cloetens *et al.*, 1999a). Once the phase maps are obtained through holographic reconstruction, there is no conceptual difficulty in bringing together many maps corresponding to different orientations of the sample, and in producing the tomographic, three-dimensional, reconstruction procedure. For each of the angular positions of the sample, the phase map is retrieved using images recorded at several (typically four) distances. The highest accessible spatial frequency is determined by the resolution of the detector. This combined quantitative phase mapping and tomography procedure, called **holotomography** (Cloetens *et al.*,1999b), provides a very useful approach to the characterization of materials on the micrometer scale.

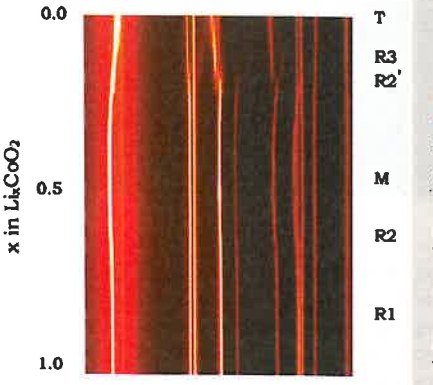
The "holotomographic" procedure was applied to an Al-Si alloy, quenched from a temperature where a "mainly Al" solid phase is surrounded by an Al-Si liquid one. This is an important industrial material because in the "semi-solid" state it is possible to give a desired shape to the alloy, this shape being retained when cooled to room temperature. The density difference between the two phases is in the 1-2% range. For each angular position of the sample 4 images were recorded at different distances, yielding 3D quantitative images that show, with a voxel edge size of 1 μm, the distribution of electron density, hence of mass density, in the sample. The highest accessible spatial frequency is determined by the maximum resolution of the detector (~1-2 μm). Fig 3 compares the possibilities of absorption microtomography (fig. 3a, where the phases are indistinguishable, and only iron-rich metallic inclusions are visible) with edge-enhancement phase microtomography (fig. 3b, where the phase boundaries are outlined by a black-white line) and with holotomography (fig. 3c, where the two phases are clearly observable through their grey level), and shows in 3d) a 3D rendering of what was the "liquid" phase.

Diffraction experiments on industrially related topics

The accessibility of hard penetrating X rays that can be focussed into sub micron spots while retaining extremely high probe intensity has opened up the field of diffraction to industrially interesting fields. New experiments with millisecond time-resolution, sub micron spatial resolution with bulk probing capability of several tens of centimetres in high Z materials can now be performed to map out stress/strain fields, to follow reactions in real time or to study *in situ* processes in complex environments such as inside reaction vessels or even in working devices like batteries or other electrochemical cells.

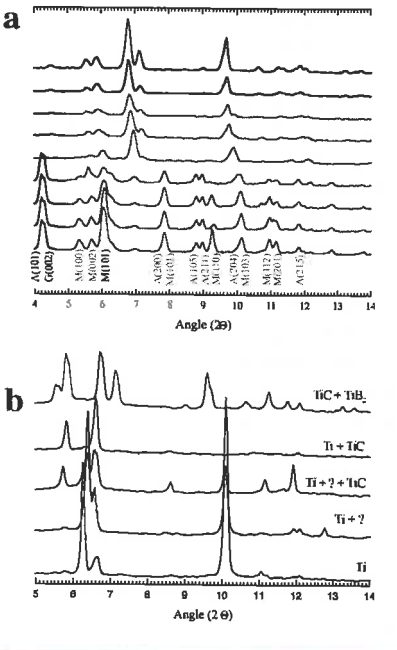
Time resolution

Time-resolved studies tend to fall into three categories: the second to several minute processes, millisecond to microsecond processes and extremely fast pico-second reactions. The last category can only be studied at present by "stroboscopic" experiments were the process is repeated many times to obtain sufficient counting statistics. Most industrial reactions however fall into the first two categories and are usually irreversible ruling out stroboscopic measurements. The present synchrotron facilities provide sufficient x-ray flux to perform one-shot experiments well down in time to the microsecond regime. The 2 dimensional diffraction data is recorded by fast read-out detectors such as large CCD cameras.

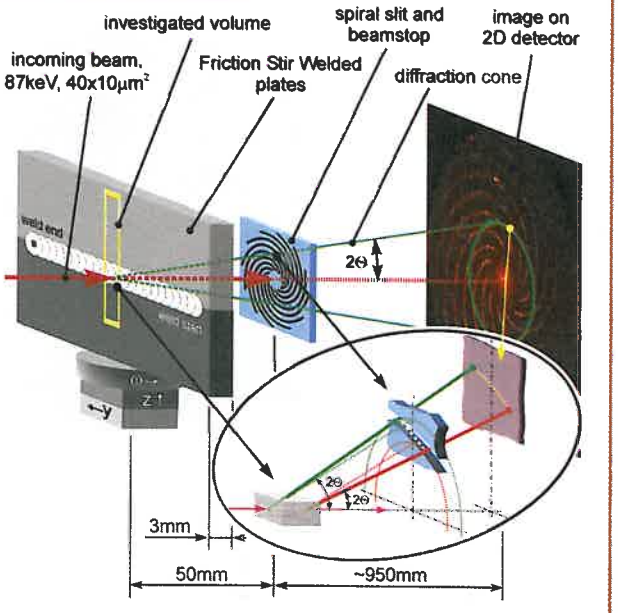


◀ **Fig. 4:** Diffraction patterns collected during *in situ* reduction of Li_xCoO_2 batteries. The letters on the right indicate the different structural phases through which the electrode material passes.

An example is given by the in-situ study of an electrochemical reaction experiment on a working Li_xCoO_2 battery (Morcrette *et al.* 2002). Strong diffraction spots from highly crystalline current collectors and packaging material have in the past compromised the weaker diffraction from the material under study. In order to de-emphasise the strong diffraction it is now possible to use micro beams and combine diffraction rapidly collected from many different areas. In the case of the Li_xCoO_2 battery 16 different orientations were collected in less than 2 secs and the pixel by pixel medians of the images collected by the CCD camera could be used to reduce the influence of the electrodes. One-dimensional powder patterns from 2 D images can thus be produced with sufficient precision to obtain accurate time-resolved behaviour of the phase fractions during the working cycle of the battery. The structures of the phases that are stable only under applied voltage



▲ **Fig. 5:** (a) The diffraction pattern during the reaction between A (anatase), G (graphite) and M (magnesium). The time resolution is 0.65 msec. (b) The diffraction patterns captured while the reaction between the metals take place. The time interval is 0.65 ms.



▲ **Fig. 6:** Schematic of the experimental setup with spiral slit and online image plate scanner on ID15B.

showed the existence of intermediate phases as well as the terminal layered CoO_2 structure. Figure 4 gives the diffraction phases during *in situ* reduction of the Li_xCoO_2 battery with the different rhombohedral, monoclinic and triclinic phases indicated.

The highest time-resolution for irreversible reactions so far (10-30ms) has been obtained in the study of self-propagating high temperature synthesis (SHS). SHS is based on the characteristic of highly exothermic reactions to sustain themselves following an initiation by relatively mild conditions. These reactions typically proceed as reaction fronts with speeds of up to 25cm/sec under temperatures up to 5000C. An example is given by Contreras *et al.* 2004 where the production of TiC and TiB_2 was studied via two routes: from elements and from oxides. The reactions were monitored by 0.2601 Å X rays *in situ*. The starting powders were compacted to cylindrical pellets and the reaction was started by external ignition at the bottom of the pellets. The X- ray beam was focused on the central part of the pellet and the reaction process was recorded as the reaction front passed the probing X-ray beam. The bulk process was followed by recording the diffraction in transmission by a CCD camera. The detailed process could be recorded and showed that the entire process took place during less than 0.1 second. The different synthesis routes produced different final microstructures that depended on the starting conditions. The particle size when pure elements were used was 10 µm whereas the reaction using oxides and Mg had sub-µm microstructure. Fig. 5 gives the diffraction patterns during the critical phases of the reactions.

Combined diffraction and imaging

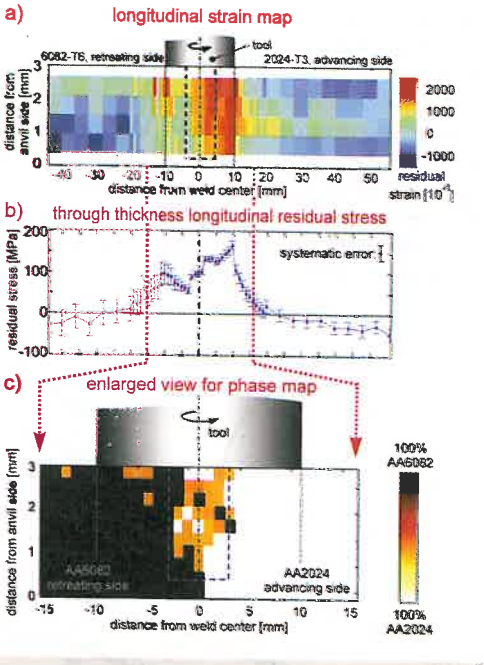
High spatial resolution

The mapping of residual stresses in components is essential in providing a roadmap for future failure modes in materials and a detailed knowledge of their distribution can indicate potential alleviating processes, which may lengthen component lifetime and avert catastrophic failures such as fractures in railroad tracks or airplane turbine blades. Until recently only destructive methods or methods with large measuring gauges have been employed. Recent developments at the high energy synchrotron facilities have now produced highly penetrating micro-focus beams with measuring gauges down to ~5*5*50 µm³. The experimental set-ups with CCD cameras or narrow receiving slits and micro-precision translation tables now give spatial resolution on the µm scale. High precision lattice parameters with $\Delta d/d \sim 10^{-6}$ can thus be determined with the small measuring gauge and excellent spatial resolution. A recent example of a novel strain and phase-mapping technique is given by Martins *et al.* 2003 in their depth resolved investigation of friction stir welding. Friction stir welding is a new method for solid welding using a spinning tool forced along a joint line. This new method can overcome problems such as weld porosity, use of filler materials and cracking in the heat-affected zone. In this study the bulk investigation of several mm thick Al plates was performed, and the depth resolution was obtained by a spiral slit assembly.

The experimental set up is illustrated in Fig. 6 and the resulting distribution of the Si phase and the residual micro strain is given in Fig. 7 for this weld of a single phase Al alloy and an $AlSi_{10}Mg$ phase.

Multi-crystal techniques

The excellent spatial resolution now available opens up entirely new opportunities for studying dynamical phenomena at the mesoscopic level. Surprisingly theoretical understanding of many fundamental materials processes, such as recrystallisation and deformation, relies on models that have built in assumptions such as sample homogeneity and absence of grain-grain interactions. The new experimental facilities can now test these assumptions. An example is given by Offerman *et al.* 2002 in their study of grain growth following phase transformations in a steel sample. Using the spatial resolving power of the synchrotron beam it was shown



▲ **Fig. 7:** (a) Depth resolved residual macrostrains in longitudinal direction (parallel to weld). (b) through thickness longitudinal residual stress component, calculated from the strain measurements in three dimensions. (c) enlarged view of the distribution of the plate material (AA6082 and AA2024) in the stirring zone.

that four different growth mechanisms were present in ferrite and these were correlated with the local environment of the grains indicating that the present models are far from adequate. Fig. 8 shows the integrated intensity of 5 grains near the cube orientation shown as a function of annealing time. At present several hundred different grains can be monitored simultaneously. This method of simultaneous determination of orientation within a bulk sample (Lauridsen *et al.* 2000) gives interesting access to the mesoscale. It is now possible, via tracking techniques, to map out grain boundaries with resolutions down to 5 μm and to follow kinetics and dynamics of groups of individual grains during processes such as heating, torsion etc.

Industrial application of Bragg diffraction imaging (X-ray topography)

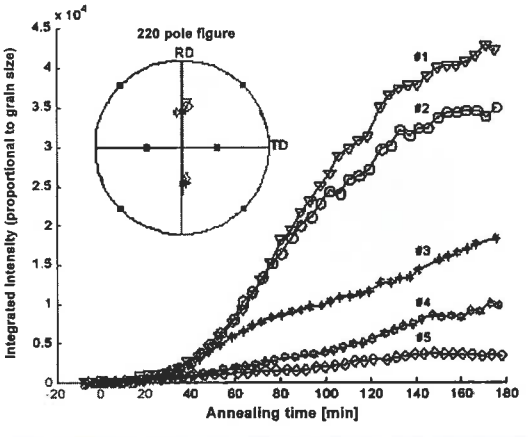
Figure 9 shows the image recorded using the 444 Bragg diffracted beam from a flux-grown platelet-shaped gallium-substituted yttrium iron garnet crystal ($Y_3Ga_xFe_{5-x}O_{12}$, with x~1, Ga-YIG). These Ga-YIG crystals are grown to produce hyper-frequency resonators. It was observed that some of these crystals exhibit spurious modes, which are very detrimental to their performance. This X-ray diffraction topographic investigation was therefore performed to identify the crystal defects present on some of the resonators, produced during growth, which are responsible for these spurious modes. Many types of defects are observable on the topographs, and one of them, the dissolution bands, appeared to be the origin, through the occurrence of "closure-like" magnetic domains, of these modes.

Future developments

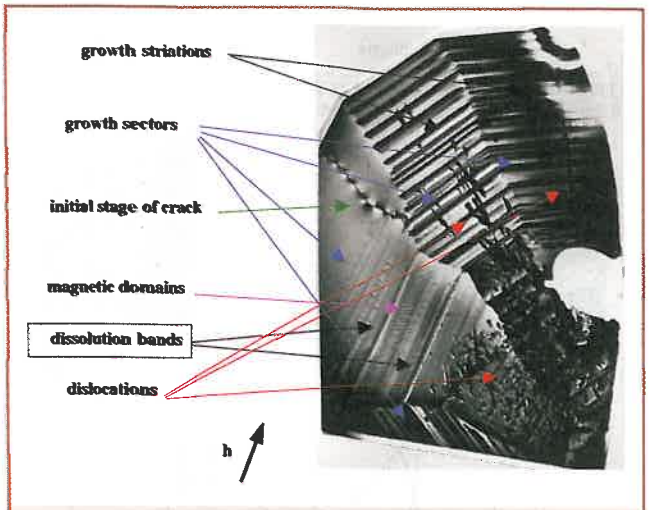
The above examples give only a few insights of possible studies of industrial phenomena using diffraction and imaging at the synchrotrons.

X-ray microtomography is an invaluable tool to obtain 3D data on a large variety of materials. The use of modern synchrotron radiation sources opens up new possibilities, with "fast tomography" to investigate the evolution of a system when varying an external parameter (time, temperature, stress, etc.) and/or improved spatial resolution, going down to the 100 nm range. In addition phase images reveal phenomena hardly visible by other means.

Other areas include the rapidly expanding field of high-pressure diffraction where micro- focussed beams and laser-heating capabilities open up new experimental routes to studies of synthesis



▲ Fig. 8: The integrated intensity of five grains near the cube in steel as a function of annealing time



▲ Fig. 9: Topograph of a flux grown Ga-YIG crystal.

and equation of states under non-ambient conditions such as megabars of pressure and several thousands of degrees of temperature.

High-resolution powder diffraction is advancing rapidly and *ab initio* structure determinations of very large structures can now be performed on materials, which cannot be obtained as single crystals.

The current trend in instrumentation development will in the near future give access to nanometre size beams. This leads to another type of image through the use of X-ray microbeam-based scanning imaging approaches, probing for instance the fluorescence or the absorption near absorption edges. The diffraction experiments will more and more be combined with complementary techniques such as imaging or Raman spectroscopy, by performing simultaneous observations. Development in detector technology will further advance the time- and space-resolving aspects of the experiments.

About the authors

Åke Kvick graduated from Uppsala University in 1974. Work on hydrogen bonded systems, zeolites and time-resolved studies with neutrons and X-rays. Scientist at Brookhaven National Lab. 1980-1989. From 1989 work at the ESRF on synchrotron radiation diffraction; presently Head of Materials Science Group and editor of J. Synch.Rad.

José Baruchel graduated in Physics at the University of Grenoble. From 1971 to 1991, work on magnetic domains and nearly perfect crystals, with X-rays and neutrons. From 1991, work at the ESRF on synchrotron radiation imaging (topography and tomography); presently Head of the X-ray Imaging Group.

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What is your strategy for Framework 6?

Sean McCarthy, Managing Director of Hyperion Ltd.

How many research organisations have a formal written strategy for Framework 6? How many individual researchers have a personal strategy for Framework 6? This article presents a checklist for the writing of a simple but practical strategy for Framework 6.

An organisation's strategy for Framework 6

The following are examples of statements of objectives that could be used in a research organisation's strategy for Framework 6. They are based on actual discussions with research organisations on their Framework 6 strategies. (The comments in brackets are actual quotations from research managers)

- To access European Union Funding for research activities. One of the problems with this simple objective is that the researchers may concentrate on obtaining any contracts rather than focussing on the research priorities of the organisation.
- To establish the research group as the European (or International) scientific leader in a scientific area ('to be the best in our field'). It is important that the scientific areas are well defined (a niche within an niche). For example, ' to be the world leader in the simulation and design of photovoltaic (solar) systems'.
- To access new technologies relevant to the organisation's areas of excellence ('to avoid missing the train').
- To work with the best research partners in the European Union. ('to be the preferred partner of the best scientists in our field')
- To provide better education and training to graduates and post-graduates. ('We want our graduates to excel in any interview, anywhere in the world')
- To promote the organisation's scientific and technical excellence to the scientific/technical community ('more conferences, more publications, more website hits...')
- To ensure the research results are used by enterprises and society ('getting value from our research efforts')
- To provide relevant support to researchers ('To streamline the process of proposal writing, contract negotiation and contract management/administration').

The strategy should also describe the activities that the organisation will NOT undertake in Framework 6. For example:

- Specific tasks in the contract. (One University clearly informed its researchers that 'we will not act as main contractor in Framework 6 contracts').
- No subcontracting. (' We will utilise only internal resources in the contracts')

A strategy for individual researchers

An individual researcher (or a small research group) should have a strategy covering all of the following issues:

- Which is the scientific niche (or even a 'niche within a niche') where the researcher's expertise is the best and where it complements that of researchers from other research centres. This requires clear identification.
- Which Framework 6 Priorities (sub-programmes) are relevant